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Mechanical Testing of TR-55 Rubber Thermally Aged Under Tensile Strain

W. Small IV, C. T. Alviso, T. S. Wilson, S. C. Chinn, R. S. Maxwell

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Mechanical Testing of TR-55 Rubber Thermally Aged Under Tensile Strain

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Part 1: Tensile Testing to Failure

Work Done By: C. Alviso (aging), W. Small (tensile testing and analysis)
Dates of Work: 3/19/08-7/7/08 (aging), 10/15/08-11/5/08 (tensile testing)
Report Written By: W. Small
Date of Report: 11/13/08, revised 2/10/09 (added engineering values), revised 2/25/09 (added strain aging information)

SUMMARY

TR-55 rubber specimens were previously subjected to an aging process consisting of the application of a tensile strain of approximately 67%, 100%, 133%, or 167% elongation for 4, 8, 12, or 16 h at either 250 °C or room temperature. Control specimens at the same temperatures/durations were not subjected to tensile strain. The specimens were allowed to recover at room temperature without external stimuli for over 100 days before tensile testing. A single dog bone was cut from each specimen and a stress-strain curve was obtained. The elastic modulus of each specimen was calculated.

Specimens aged under tensile strain exhibited rubber-like behavior dependent on the aging elongation and duration. This behavior was not evident in the unstrained controls. For the unstrained controls, exposure to 250 °C resulted in an increase in modulus relative to the unheated material independent of the heating duration. The tensile strain applied during the aging process caused a reduction in modulus relative to the controls; lower moduli were observed for the shorter aging durations. Slippage of the specimens in the grips prevented determination of ultimate strength, as all specimens either slipped completely out of the grip before failure or failed at the original grip edge after slipping.

METHODS

The TR-55 material was made at Kansas City Plant. TR-55 rubber specimens were previously subjected to an aging process consisting of the application of a tensile strain of approximately 67%, 100%, 133%, or 167% elongation for 4, 8, 12, or 16 h at either 250 °C or room temperature (see Appendix A). Control specimens at the same temperatures/durations were not subjected to tensile strain. The specimens were allowed to recover at room temperature without external stimuli for over 100 days before tensile testing.

A single dog bone was cut from each specimen using an ASTM D-638-V die immediately before tensile testing. The width and length of the narrow section were 3.18 mm and 9.53 mm, respectively. Specimen thickness was between 0.516 and 0.736 mm (measured in the narrow section using a micrometer). Specimens were secured in the tensile tester grips (initial grip separation ≈ 10 mm) and stretched at a rate of 10 mm/min (strain rate ≈ 1 min^{-1}). True stress was calculated from the output of a 50-N load cell. True strain was calculated from the crosshead position. All tests were performed at room temperature in B132S R2729.

Simply securing the specimen in the grips resulted in a compressive load as the grips were tightened. To eliminate this initial compression of the specimen, the following procedure was performed. First, the grip separation was set to 10 mm. Second, the specimen was secured in the movable upper grip only. Third, the upper grip was lowered ~1.0 mm. Finally, the specimen was secured in the fixed lower grip, and the upper grip was raised until the load returned to 0±0.010 N. The grip separation was generally slightly greater than 10 mm following this procedure (i.e., the initial strain was slightly greater than zero).

The software automatically reduced the data acquisition rate from the originally specified value of 10 Hz to 1.2-2.5 Hz due to the relatively long test durations. This was not discovered until all tests were completed. The first data point was generally acquired at $t \approx 1$ s.

The raw data was corrected to compensate for the nonzero strain at the initial time. Normally this would be accomplished by adding the initial extension value to the original grip separation value (i.e., original specimen length) in the true stress and true strain calculations, yielding a corrected initial strain value of zero. However, due to the slow data acquisition rate relative to the extension rate, a new initial data point was manually inserted into the raw data at t=0.5 s. The choice of 0.5 s, though somewhat arbitrary, was based on unrelated data acquired at a rate of ~10 Hz using the same tensile tester. The extension at this time was calculated by subtracting the distance the crosshead moved in the time between t_0=0.5 s and the time t_1 of the first acquired data point (~1 s) from the recorded extension at the first acquired data point. This distance is given by $s(t_1-t_0)$, where s is the extension rate in mm/s (10 mm/min = 0.17 mm/s). This new initial extension value was used as the basis for the correction. The load at the newly inserted initial data point was assumed to be zero (not used in any calculations).

The elastic modulus was reported as the maximum slope of the true stress-strain curve below a true strain of 0.05. Because it was artificially generated, the (0,0) data point was not used in the calculation of the modulus.

RESULTS

Representative true stress-strain curves are shown in Figs. 1 and 2 (engineering stress-strain curves are shown in Figs. B1 and B2 in Appendix B). Each plot includes a curve corresponding to the virgin (unheated, unstrained) material for reference. Data beyond true strain values of 1.4 are not shown, as slippage of the specimens in the grips generally began to occur at a true strain of ~1. Periodic yielding was evident as the specimen slipped out of the grip, tooth by tooth.

Specimens aged under tensile strain exhibited rubber-like behavior characterized by an initial stress increase followed by a stress plateau and a terminal stress increase. The unstrained controls did not exhibit a stress plateau. The onset of the terminal stress increase depended on the aging elongation and duration.

The elastic modulus of each specimen is given in Table 1. The data is shown graphically in Fig. 3. For the unstrained controls, exposure to 250 °C resulted in an increase in modulus relative to the unheated (virgin) material independent of the heating duration. The tensile strain applied during the aging process caused a reduction in modulus relative to the controls; lower moduli were observed for the shorter aging durations.

Teeth marks left by the grip were visible on the specimens following tensile testing. All specimens either failed at the original grip edge (indicated by the teeth marks on the specimen) after slipping partially out of the grip or slipped completely out of the grip before failure. Therefore, ultimate strength was not determined.

Table 1: Elastic Moduli of TR-55 Specimens Thermally Aged Under Tensile Strain

	4 h, 250 °C		8 h, 250 °C		12 h, 250 °C		16 h, 250 °C		6 days, Room Temp	
Elongation	**Aging Date**	**E (MPa)**	**Aging Date**	**E (MPa)**	**Aging Date**	**E (MPa)**	**Aging Date**	**E (MPa)**	**Aging Date**	**E (MPa)**
0%	4/22/2008	5.01	7/1/2008	5.18	7/7/2008	4.66	4/22/2008	4.91	NA	4.05‡
(Control)	4/17/2008	5.11	4/9/2008	4.79	4/21/2008	4.99	4/16/2008	4.74	NA	4.31‡
	4/11/2008	5.07					4/3/2008	5.07	NA	4.05‡
	4/10/2008	3.32*†								
	4/2/2008	4.81								
	Mean	*5.00*	*Mean*	*4.99*	*Mean*	*4.83*	*Mean*	*4.91*	*Mean*	*4.14‡*
	StDev	*0.13*	*StDev*	*0.28*	*StDev*	*0.23*	*StDev*	*0.17*	*StDev*	*0.15‡*
67%	4/22/2008	3.29	7/1/2008	3.25	7/7/2008	3.59	4/22/2008	3.27	3/19/2008	3.82
	4/17/2008	3.39	4/9/2008	3.27	4/21/2008	3.30	4/16/2008	3.90		
	4/11/2008	3.48					4/3/2008	4.26		
	4/10/2008	3.32								
	4/2/2008	3.37								
	Mean	*3.37*	*Mean*	*3.26*	*Mean*	*3.45*	*Mean*	*3.81*	*Mean*	*3.82*
	StDev	*0.07*	*StDev*	*0.01*	*StDev*	*0.21*	*StDev*	*0.50*	*StDev*	*NA*
100%	4/22/2008	3.17	7/1/2008	3.12	7/7/2008	3.65	4/22/2008	3.84	3/19/2008	3.45
	4/17/2008	3.18	4/9/2008	3.19	4/21/2008	3.24	4/16/2008	3.71		
	4/11/2008	3.09					4/3/2008	4.02		
	4/10/2008	3.33								
	4/2/2008	3.32								
	Mean	*3.22*	*Mean*	*3.16*	*Mean*	*3.45*	*Mean*	*3.86*	*Mean*	*3.45*
	StDev	*0.10*	*StDev*	*0.05*	*StDev*	*0.29*	*StDev*	*0.16*	*StDev*	*NA*
133%	4/22/2008	3.04	7/1/2008	3.16	7/7/2008	4.03	4/22/2008	3.75	3/19/2008	3.61
	4/17/2008	3.97*	4/9/2008	3.21	4/21/2008	3.99	4/16/2008	3.87		
	4/11/2008	2.98					4/3/2008	3.93		
	4/10/2008	3.08								
	4/2/2008	3.04								
	Mean	*3.04*	*Mean*	*3.19*	*Mean*	*4.01*	*Mean*	*3.85*	*Mean*	*3.61*
	StDev	*0.04*	*StDev*	*0.04*	*StDev*	*0.03*	*StDev*	*0.09*	*StDev*	*NA*
167%	4/22/2008	3.32	7/1/2008	3.35	7/7/2008	3.56	4/22/2008	3.46	3/19/2008	3.28*†
	4/17/2008	3.05	4/9/2008	3.48	4/21/2008	3.63	4/16/2008	3.76		
	4/11/2008	3.13					4/3/2008	3.88		
	4/10/2008	3.18								
	4/2/2008	3.00								
	Mean	*3.14*	*Mean*	*3.42*	*Mean*	*3.60*	*Mean*	*3.70*	*Mean*	*NA*
	StDev	*0.13*	*StDev*	*0.09*	*StDev*	*0.05*	*StDev*	*0.21*	*StDev*	*NA*

*Data excluded from mean
†Invalid test specimen
‡Virgin material

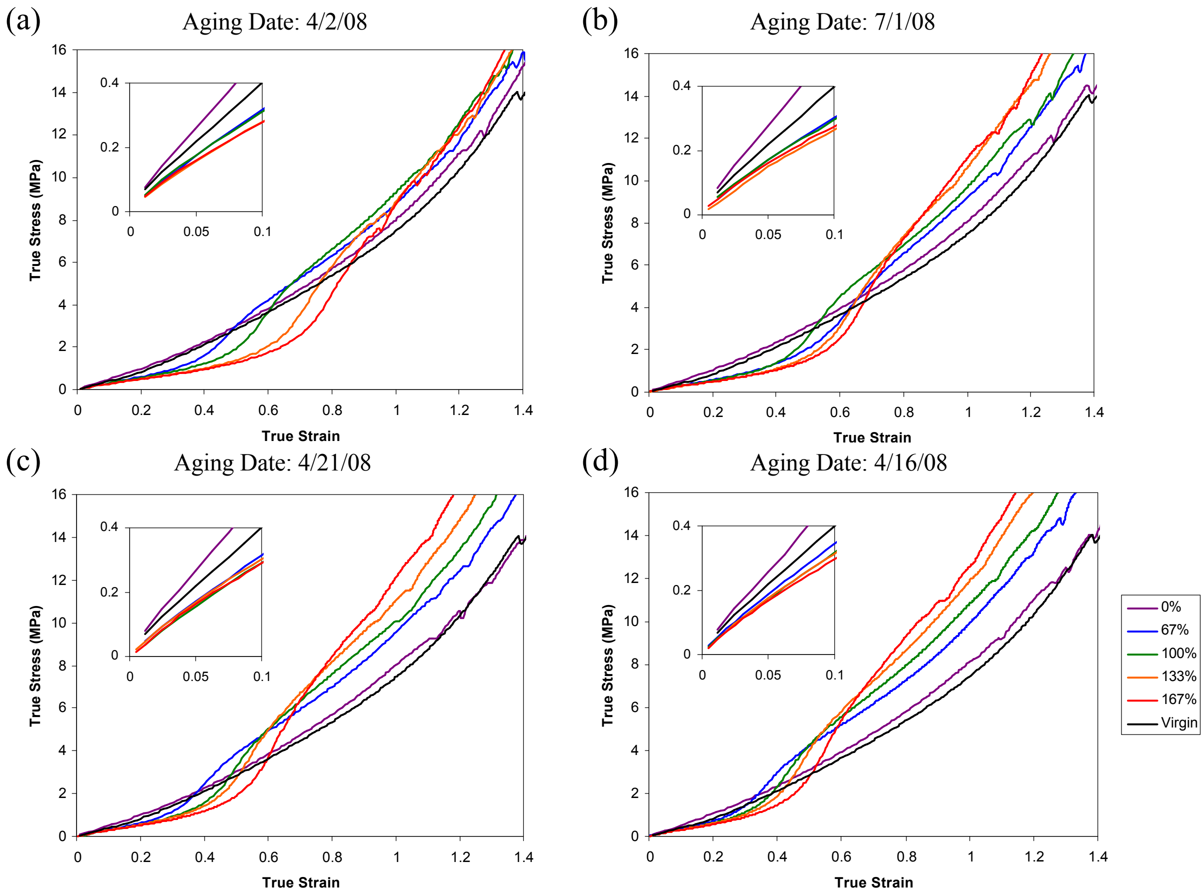

Fig. 1. True stress-strain curves of TR-55 specimens thermally aged at 250 °C under tensile strains of 0%, 67%, 100%, 133%, or 167% elongation for (a) 4 h, (b) 8 h, (c) 12 h, and (d) 16 h. A curve representing the virgin material is included in each plot. The region of low stress/strain is shown in the inset of each plot (same units as main plot).

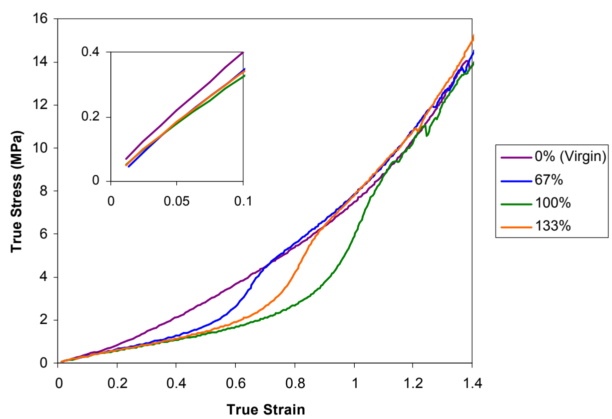

Fig. 2. True stress-strain curves of TR-55 specimens aged for 6 days at room temperature under tensile strains of 0%, 67%, 100%, or 133% elongation (the tensile test specimen aged under 167% elongation was not valid and is therefore not shown). The region of low stress/strain is shown in the inset (same units as main plot).

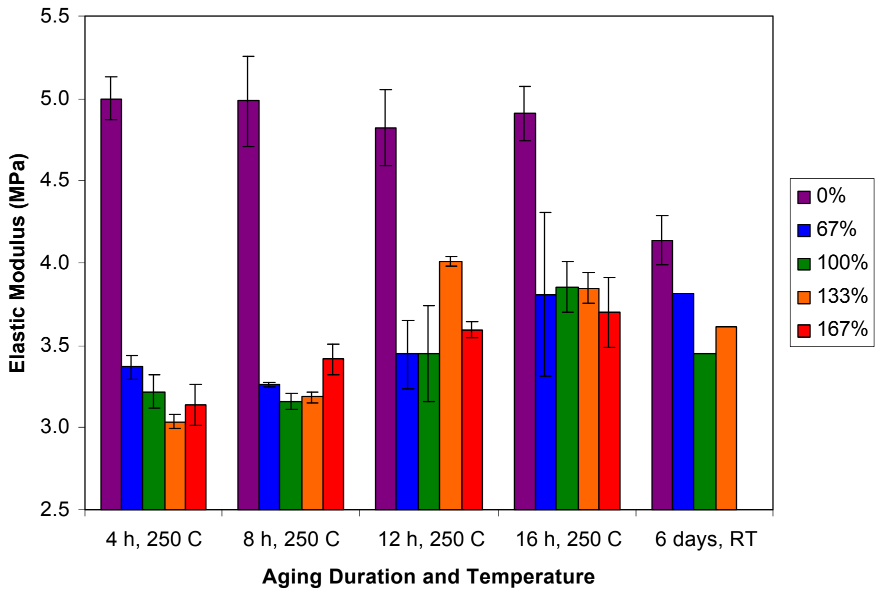

Fig. 3. Elastic moduli of TR-55 specimens aged under tensile strains of 0%, 67%, 100%, 133% or 167% elongation at 250 °C for 4, 8, 12, or 16 h or room temperature (RT) for 6 days. The columns represent mean values and the error bars represent the standard deviation when n>1 (see Table 1).

APPENDIX A
Specimen Stretching in the Aging Process

The stretching fixture consisted of two movable clamps on a base plate with preset pins to determine the separation between the clamps. With the clamps spaced 1.5" apart (see Fig. A1), a rectangular strip (~10 mm wide and <1 mm thick) of TR-55 was secured in the fixture (i.e., original specimen length = 1.5"). To stretch the specimen, the separation between the clamps was increased to one of four distances:

#1: 2.5" (67% elongation)
#2: 3.0" (100% elongation)
#3: 3.5" (133% elongation)
#4: 4.0" (167% elongation)

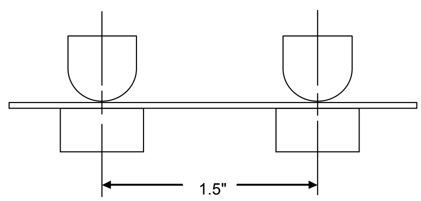

Fig. A1. Schematic of a specimen clamped in its initial pre-stretched position.

To determine the extent of specimen slippage in the clamps (before heating), the stretched length was calculated for each specimen based on the measured change in specimen width according to Chinn et al., Polymer Degradation and Stability 2006; 91:555-64 (Table A1).

Table A1: Calculated Specimen Length in Stretched Position

Target	4 h, 250 °C		8 h, 250 °C		12 h, 250 °C		16 h, 250 °C		6 days, Room Temp	
Length	Aging Date	Length	Aging Date	Length	Aging Date	Length	Aging Date	Dist.	Aging Date	Length
#1: 2.5"	4/22/2008	2.54"	7/1/2008	2.56"	7/7/2008	2.59"	4/22/2008	2.56"	3/19/2008	2.39"
	4/17/2008	2.42"	4/9/2008	2.45"	4/21/2008	2.55"	4/16/2008	2.55"		
	4/11/2008	2.44"					4/3/2008	2.43"		
	4/10/2008	2.38"								
	4/2/2008	2.47"								
#2: 3.0"	4/22/2008	3.08"	7/1/2008	2.86"	7/7/2008	3.05"	4/22/2008	3.05"	3/19/2008	2.61"
	4/17/2008	2.92"	4/9/2008	2.83"	4/21/2008	3.11"	4/16/2008	2.98"		
	4/11/2008	2.92"					4/3/2008	2.81"		
	4/10/2008	2.76"								
	4/2/2008	2.87"								
#3: 3.5"	4/22/2008	3.38"	7/1/2008	3.54"	7/7/2008	3.47"	4/22/2008	3.58"	3/19/2008	3.32"
	4/17/2008	3.42"	4/9/2008	2.98"	4/21/2008	3.45"	4/16/2008	3.35"		
	4/11/2008	3.03"					4/3/2008	3.27"		
	4/10/2008	3.00"								
	4/2/2008	3.34"								
#4: 4.0"	4/22/2008	3.98"	7/1/2008	4.00"	7/7/2008	3.79"	4/22/2008	3.97"	3/19/2008	3.49"
	4/17/2008	3.76"	4/9/2008	3.36"	4/21/2008	3.94"	4/16/2008	3.80"		
	4/11/2008	3.51"					4/3/2008	3.13"		
	4/10/2008	3.13"								
	4/2/2008	3.72"								

APPENDIX B
Engineering Values

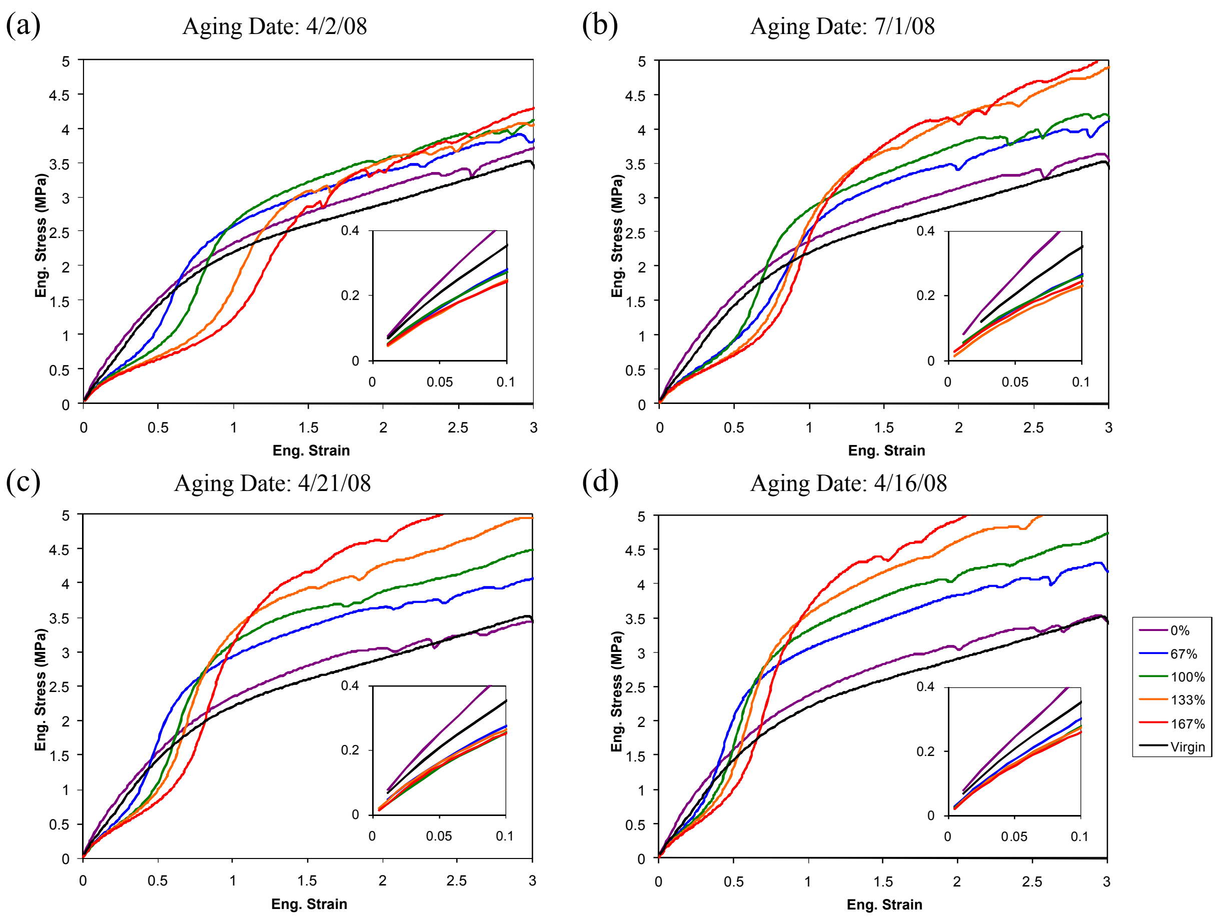

Fig. B1. Engineering stress-strain curves of TR-55 specimens thermally aged at 250 °C under tensile strains of 0%, 67%, 100%, 133%, or 167% elongation for (a) 4 h, (b) 8 h, (c) 12 h, and (d) 16 h. A curve representing the virgin material is included in each plot. The region of low stress/strain is shown in the inset of each plot (same units as main plot).

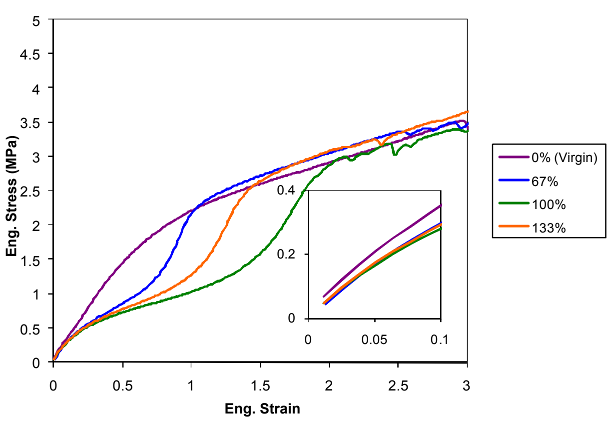

Fig. B2. Engineering stress-strain curves of TR-55 specimens aged for 6 days at room temperature under tensile strains of 0%, 67%, 100%, or 133% elongation (the tensile test specimen aged under 167% elongation was not valid and is therefore not shown). The region of low stress/strain is shown in the inset (same units as main plot).

Part 2: Cyclic Tensile Testing

Work Done By: C. Alviso (aging), W. Small (tensile testing and analysis)
Dates of Work: 1/13/09-1/14/09 (aging), 1/15/09-1/16/09 (tensile testing)
Report Written By: W. Small
Date of Report: 2/18/09

SUMMARY

TR-55 rubber samples were previously subjected to an aging process consisting of the application of a tensile strain of approximately 100% or 167% elongation for 4 or 16 h at 250°C (procedure described in Part 1). Control specimens were heated, but not subjected to tensile strain. The samples were allowed to recover at room temperature without external stimuli before cyclic tensile testing. Two rectangular specimens were cut from each sample. Each specimen was subjected to five load/unload cycles and a stress-strain curve was obtained. The elastic modulus of each specimen was calculated.

Specimens aged under tensile strain exhibited rubber-like behavior dependent on the aging elongation and duration during all cycles. This behavior was not evident in the unstrained controls during the first cycle, but was apparent in subsequent cycles. All specimens showed a large hysteresis loss during the first cycle. Subsequent cycles showed behavior similar to the first unloading segment with much smaller hysteresis losses. The peak load stress decreased with each cycle for all specimens. Residual plastic deformation was evident. For the unstrained controls, exposure to 250°C resulted in an increase in modulus relative to the unheated material independent of the heating duration. The tensile strain applied during the aging process caused a reduction in modulus relative to the controls.

METHODS

The TR-55 material was made at Kansas City Plant. TR-55 rubber samples were previously subjected to an aging process consisting of the application of a tensile strain of approximately 100% or 167% elongation for 4 or 16 h at 250°C. Control specimens were heated, but not subjected to tensile strain. The samples were allowed to recover at room temperature without external stimuli before cyclic tensile testing.

Two rectangular specimens, each approximately 30 mm long, were cut from each sample using a scalpel immediately before cyclic tensile testing. Specimen width was between 3.14 and 3.83 mm, not including one specimen with a width of 4.60 mm that slipped out of the tensile tester grip during the loading segment of the first cycle. Specimen thickness was between 0.521 and 0.695 mm. Width and thickness measurements were made using a micrometer. Specimens were secured in the grips (initial grip separation ≈ 20 mm) and stretched at a rate of 20 mm/min (strain rate ≈ 1 min^{-1}). Each specimen was subjected to five load/unload cycles. The loading segment stretched the specimen to an engineering strain of 2.2 and held it there for 5 s. The unloading segment returned the specimen to the original starting position and held it there for 5 s. Data was acquired at a rate of 2 Hz. Stress was calculated from the output of a 50-N load cell. Strain was calculated from the crosshead position. All tests were performed at room temperature in B132S R2729.

Simply securing the specimen in the grips resulted in a compressive load as the grips were tightened. To eliminate this initial compression of the specimen, the following procedure was performed. First, the grip separation was set to 20 mm. Second, the specimen was secured in the movable upper grip only. Third, the upper grip was lowered ~0.3 mm. Finally, the specimen was secured in the fixed lower grip, and the upper grip was raised until the load returned to 0±0.010 N. The grip separation was slightly different from 20 mm (generally within ±0.050 mm) following this procedure (i.e., the initial strain was slightly different from zero).

For the elastic modulus calculation, which was based on the calculated true stress and true strain values, the raw data was corrected to compensate for the nonzero strain at the initial time. This was accomplished by adding the initial extension value to the original grip separation value (i.e., original specimen length) in the true stress and true strain calculations, yielding a corrected initial strain value of zero. Because the actual initial extension value at t=0 was not recorded in the data file, the value was recorded manually after loading the specimen. The load at the newly inserted initial data point was assumed to be exactly zero (not used in any calculations).

The elastic modulus was reported as the maximum slope of the true stress-strain curve of the loading segment of the first cycle below a true strain of 0.05. The inserted (0,0) data point was not used in the calculation of the modulus.

RESULTS

True stress and strain during all five cycles are plotted versus time in Fig. 1. Good agreement existed between both specimens from each sample (not shown). True stress-strain curves from the loading segment of the first cycle are shown in Fig. 2. Each plot includes a curve corresponding to the virgin (unheated, unstrained) material for reference. Individual true stress-strain curves including all five cycles for specimens from each aging protocol, including the virgin material, are shown in Fig. 3. The same plots using engineering values of stress and strain are shown in Figs. A1-A3 in Appendix A.

Specimens aged under tensile strain exhibited rubber-like behavior dependent on the aging elongation and duration during all cycles. This behavior was not evident in the unstrained controls during the first cycle, but was apparent in subsequent cycles. All specimens showed a large hysteresis loss during the first cycle. Subsequent cycles showed behavior similar to the first unloading segment with much smaller hysteresis losses. The peak load stress decreased with each cycle for all specimens. Residual plastic deformation was evident.

The elastic modulus of each specimen is given in Table 1. The data is shown graphically in Fig. 4. For the unstrained controls, exposure to 250°C resulted in a slight increase in modulus relative to the unheated (virgin) material. The tensile strain applied during the aging process caused a reduction in modulus relative to the controls. Though the values are somewhat different, trends are similar to those noted in the previous TR-55 tensile testing study (Part 1). Reasons for the differences are not clear.

Table 1: Elastic Moduli of TR-55 Specimens Thermally Aged Under Tensile Strain

	4 h, 250°C			16 h, 250°C			Room Temp		
Elongation	**Specimen**	**E (MPa)**		**Specimen**	**E (MPa)**		**Specimen**	**E (MPa)**	
0%	1	4.77		1	4.45		1	4.41‡	
(Control)	2	4.81		2	4.85		2	4.47‡	
	Mean	*4.79*	*(5.00*)*	*Mean*	*4.65*	*(4.91*)*	*Mean*	*4.44‡*	*(4.14*)*
	StDev	*0.03*	*(0.13*)*	*StDev*	*0.28*	*(0.17*)*	*StDev*	*0.04‡*	*(0.15*)*
100%	1	2.81		1	2.68		1	---	
	2	3.02		2	2.70		2	---	
	Mean	*2.92*	*(3.22*)*	*Mean*	*2.69*	*(3.86*)*	*Mean*	---	*(3.45*)*
	StDev	*0.15*	*(0.10*)*	*StDev*	*0.01*	*(0.16*)*	*StDev*	---	*(NA*)*
167%	1	2.90		1	3.14		1	---	
	2	2.91		2	3.32		2	---	
	Mean	*2.91*	*(3.14*)*	*Mean*	*3.23*	*(3.70*)*	*Mean*	---	*(---*)*
	StDev	*0.01*	*(0.13*)*	*StDev*	*0.13*	*(0.21*)*	*StDev*	---	*(---*)*

*Data from previous TR-55 tensile testing study (Part 1)
‡Virgin material
---Data not obtained

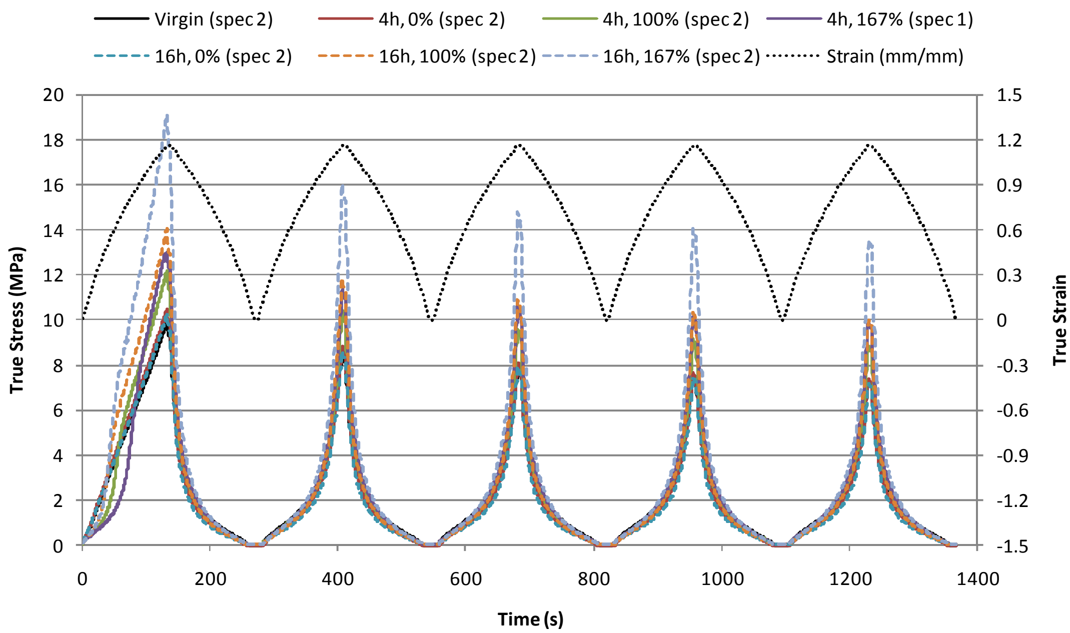

Fig. 1. True stress and strain versus time for TR-55 specimens thermally aged at 250°C under tensile strains of 0%, 100%, or 167% elongation for 4 or 16 h. A curve representing the virgin material is also shown.

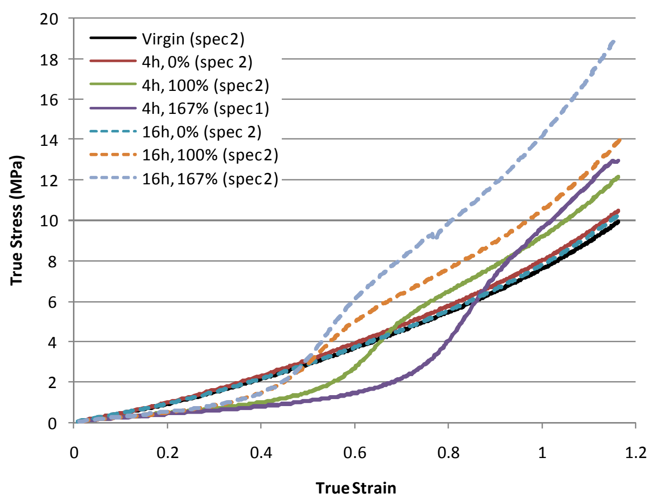

Fig. 2. True stress-strain curves from the loading segment of the first cycle for TR-55 specimens thermally aged at 250°C under tensile strains of 0%, 100%, or 167% elongation for 4 or 16 h. A curve representing the virgin material is also shown.

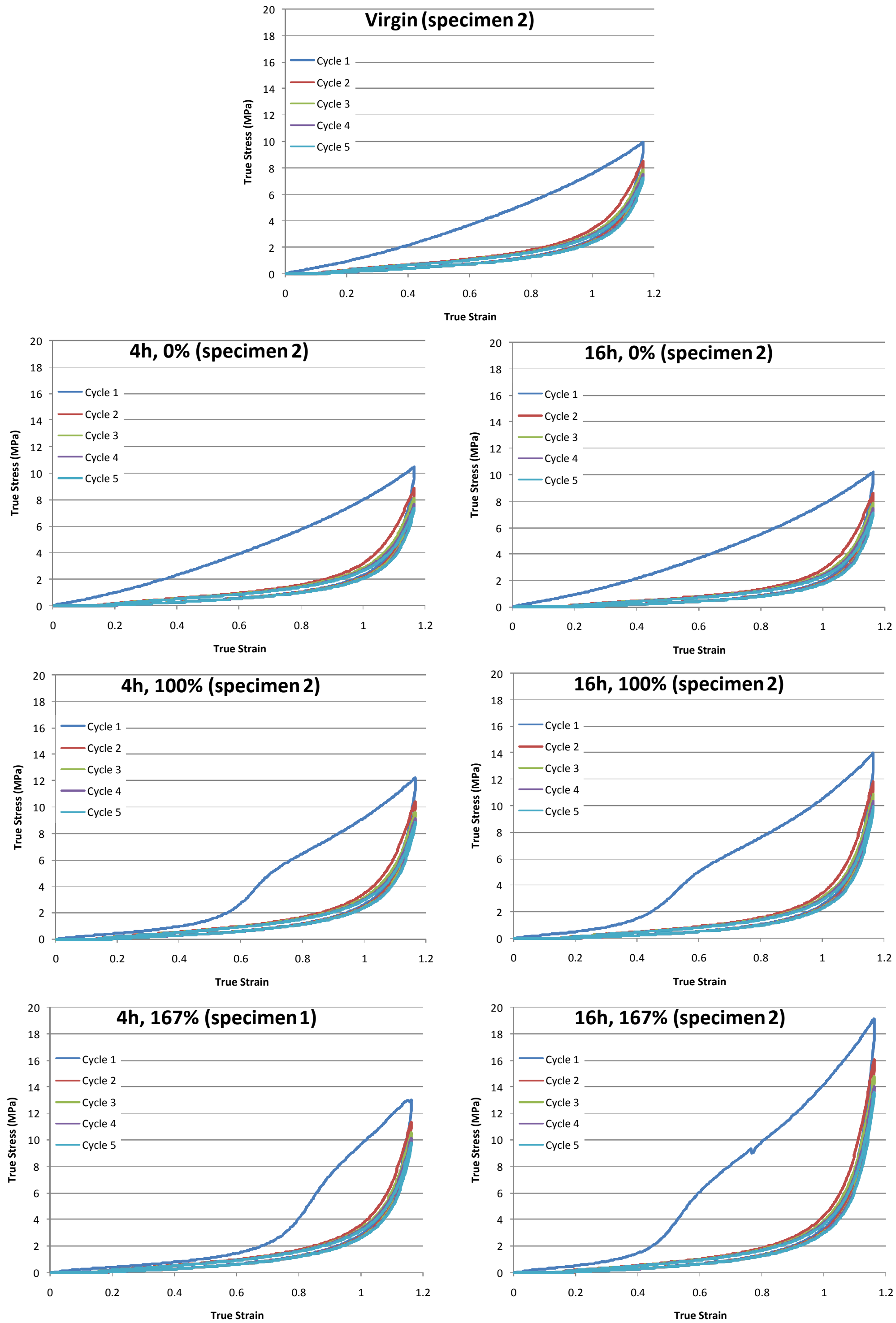

Fig. 3. True stress-strain curves including all five cycles for TR-55 specimens thermally aged at 250°C under tensile strains of 0%, 100%, or 167% elongation for 4 or 16 h. A curve representing the virgin material is also shown.

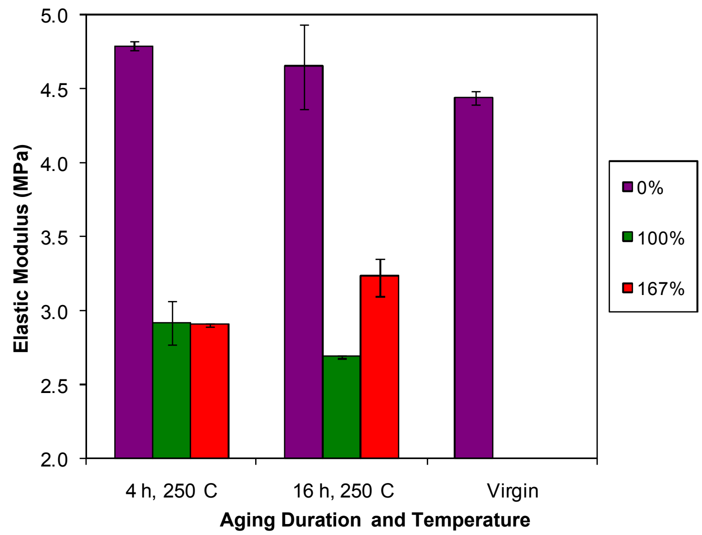

Fig. 4. Elastic moduli of TR-55 specimens aged under tensile strains of 0%, 100%, or 167% elongation at 250°C for 4 or 16 h. The modulus of the virgin material is also shown. The columns represent mean values and the error bars represent the standard deviation (see Table 1).

APPENDIX A
Engineering Values

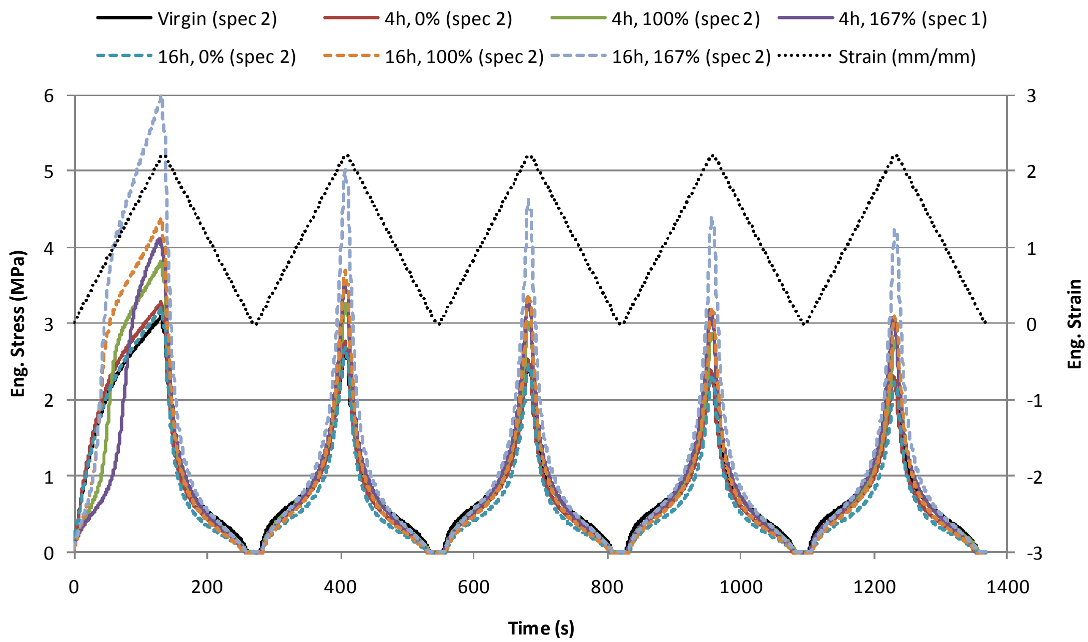

Fig. A1. Engineering stress and strain versus time for TR-55 specimens thermally aged at 250°C under tensile strains of 0%, 100%, or 167% elongation for 4 or 16 h. A curve representing the virgin material is also shown.

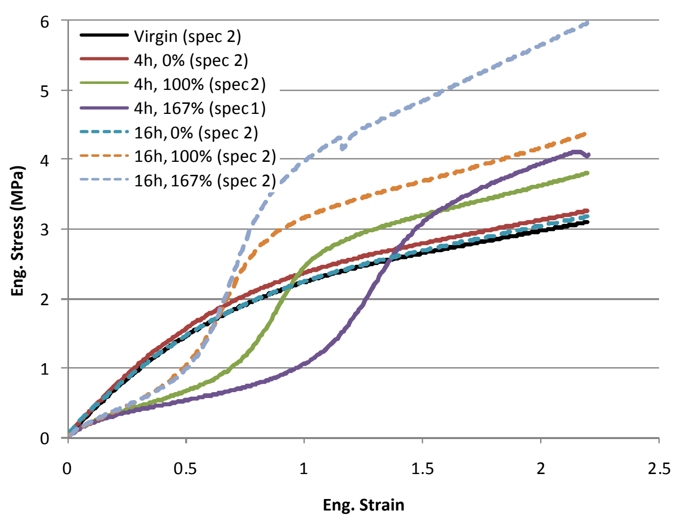

Fig. A2. Engineering stress-strain curves from the loading segment of the first cycle for TR-55 specimens thermally aged at 250°C under tensile strains of 0%, 100%, or 167% elongation for 4 or 16 h. A curve representing the virgin material is also shown.

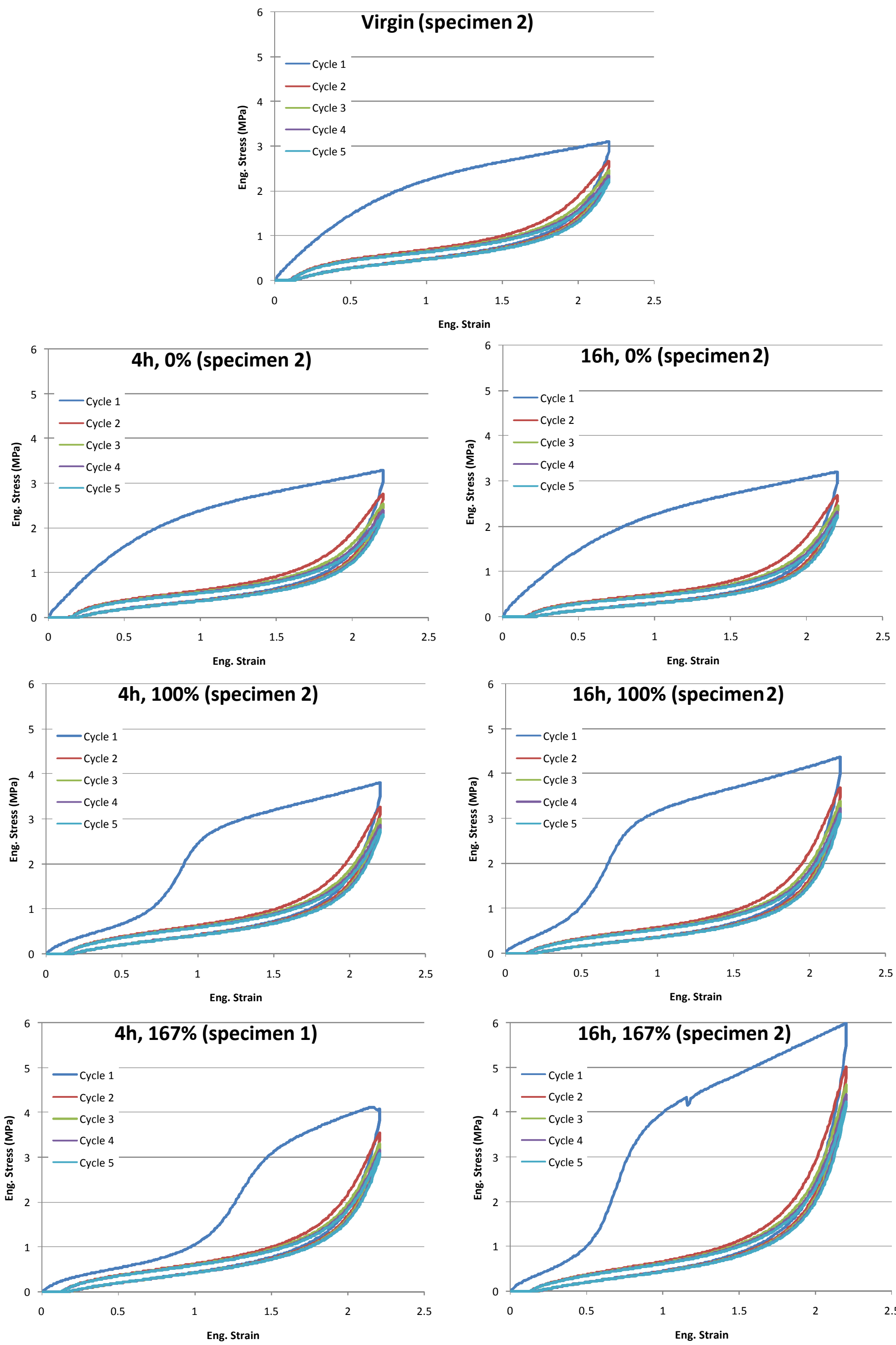

Fig. A3. Engineering stress-strain curves including all five cycles for TR-55 specimens thermally aged at 250°C under tensile strains of 0%, 100%, or 167% elongation for 4 or 16 h. A curve representing the virgin material is also shown.